



Department of Energy
Carlsbad Field Office
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July 17, 2003



Dr. Amrit S. Boparai
Analytical Chemistry Laboratory
Argonne National Laboratory-East
9700 South Cass Avenue
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RE: Transmittal of Audit Report for Audit A-03-18

Dear Dr. Boparai:

The Carlsbad Field Office (CBFO) performed Audit A-03-18 of Argonne National Laboratory-East, on June 24 and 25, 2003. The audit team concluded that the overall status of the ANL-E Quality Assurance Program in its role as the sample preparation contractor for the Headspace Gas Performance Demonstration Program is adequate, satisfactorily implemented, and effective.

The details of the audit as well as conclusions are detailed within the enclosed audit report.

If you have any questions or comments, please contact me at (505) 234-7442.

Sincerely,

M. Lea Chism
Quality Assurance Specialist

Enclosure



Dr. Amrit S. Boparai

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July 16, 2003

cc: w/enclosure

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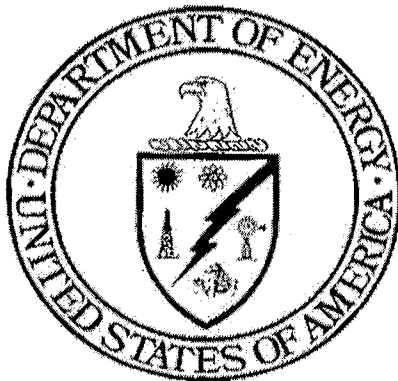
U.S. DEPARTMENT OF ENERGY
CARLSBAD FIELD OFFICE

AUDIT REPORT
OF THE
ARGONNE NATIONAL LABORATORY
CHEMICAL TECHNOLOGY DIVISION
(ANL-E)

Chicago, Illinois

AUDIT NUMBER A-03-18

JUNE 24 - 25, 2003



SAMPLE PREPARATION SUPPORT FOR HEADSPACE GAS
PERFORMANCE DEMONSTRATION PROGRAM

Prepared by: Date: Amelia L. Arceo
Amelia L. Arceo
Audit Team Leader

Date: 7/11/03

Approved for Issue by: Ava L. Holland
Ava L. Holland
CBFO Quality Assurance Manager

Date: 7/17/03

1.0 EXECUTIVE SUMMARY

CBFO Audit A-03-18 was conducted to evaluate the adequacy, implementation, and effectiveness of the Argonne National Laboratory (ANL-E) quality assurance and technical support activities for the preparation of samples needed to support the WIPP Headspace Gas (HSG) Performance Demonstration Program (PDP). The organizational structure and compliance with the procedures covering document control, quality assurance records, procurement, training and qualification, laboratory notebooks, analytical sample preparation, PDP sample verification analyses, gas standards preparation, preparation and distribution of PDP samples, and canister cleaning activities were assessed for compliance with the Quality Assurance Program Document (CAO-94-1012) Revision 5, and the PDP Plan for Analysis of Simulated Headspace Gas (DOE/CAO-95-1076), Revision 4.

The audit was conducted at the Argonne National Laboratory (ANL) Chemical Technology Division (CMT) Analytical Chemistry Laboratory (ACL) in Argonne, Illinois on June 24 - 25, 2001. The audit team determined that the activities evaluated relating to quality assurance were adequate, satisfactorily implemented and effective. The audit team determined that the HSG PDP implementing procedures and activities for sample preparation, verification and distribution were adequate, satisfactorily implemented and effective. The audit team identified 17 concerns, six of which were corrected during the audit (CDA) since they were isolated in nature and required only remedial corrective actions. Four corrective action reports (CARs) were issued concerning the use of expired standards to prepare calibration standards (CAR 03-065), four procedure adequacy issues against SOP: ACL-168 (CAR 03-066), the lack of canister inventory and "received by" statement on the certificate of analysis (CAR 03-067), and the use of a pressure gauge that reads in millimeters of mercury (mm Hg) and water that is produced using the NANO pure water system instead of those specified in SOP: ACL-158 (CAR 03-068). Two observations were issued as a result of this audit. One recommendation was provided for management consideration. The CARs, CDAs, Observations, and Recommendation are described in Section 6.0 of this report.

2.0 SCOPE

CBFO Audit A-03-18 was conducted to evaluate the adequacy, implementation, and effectiveness of procedures and activities used by the ANL CMT ACL for the preparation of samples for the HSG PDP. The procedures covering the processes used in this role, and those for the procurement of the gas standards from which the PDP samples are prepared, were included.

3.0 AUDIT TEAM, AND OBSERVER

CBFO AUDIT TEAM

Amelia I. Arceo
Cliff Watkins
Dorothy Gill

Audit Team Leader, CTAC
Technical Specialist, CTAC
Technical Specialist, CTAC

OBSERVER

Ben Walker Observer, Environmental Evaluation Group (EEG)

4.0 AUDIT PARTICIPANTS

A list of personnel contacted during the course of the audit is provided as Attachment 1 of this report.

5.0 SUMMARY OF AUDIT RESULTS

5.1 Audit Activities

Details of audit activities, along with the specific objective evidence reviewed and the results of the reviews are contained within the audit checklists. The checklists are maintained as QA records.

The audit team verified ANL CMT ACL compliance with the PDP Plan for Analysis of Simulated Headspace Gas, DOE/CAO-95-1076, Revision 4. The audit team determined that the ANL CMT ACL *Work Plan for HSG PDP Sample Preparation, Verification, and Distribution* adequately defined the current organizational structure of the project. As a result, the audit team concluded that the related QA program requirements, procedures, and activities were adequate, satisfactorily implemented, and effective. In addition, the audit team evaluated the HSG PDP sample preparation, verification analyses, canister cleaning, gas standards preparation, laboratory notebooks, quality assurance records, employee training, and the procurement of gas standards for the HSG PDP and concluded that these activities were adequate, satisfactorily implemented, and effective.

5.1.1 Organization

The audit team evaluated the adequacy of documents depicting the ANL CMT ACL organization. The ANL CMT ACL *Work Plan for Headspace Gas Performance Demonstration Program (PDP) Sample Preparation, Verification and Distribution* adequately depict the current organizational structure and define

the responsibilities of the ANL personnel involved in PDP sample preparation efforts.

5.1.2 Work Plan Implementation

The ANL CMT ACL prepared a Work Plan titled *Work Plan for Headspace Gas (HSG) Performance Demonstration Program (PDP) Sample Preparation, Verification and Distribution* to meet the applicable requirements of the Quality Assurance Program Document (CAO-94-1012), Revision 5, and the PDP Plan for Analysis of Simulated Headspace Gas (DOE/CAO-95-1076), Revision 4. Activities related to the implementation of the Work Plan were reviewed, including the Standard Operating Procedures, Procurement, Training and Qualification, and QA Records. Two concerns were identified which became an observation and CDA. The Work Plan implies that all personnel involved in the HSG PDP activities read all of the documents that are listed in the Attachment 1 of the Work Plan. Only the Technical Lead for the ANL-E HSG PDP activities has read the QAPD and the HSG PDP Plan. Because the specific training requirements are not delineated in any other documentation an observation (Observation #1) was provided for management consideration that the language of the Work Plan be made more explicit as to the training requirements for each employee. Furthermore, the review of Cycle 17A procurement documentation revealed that there was no technical specification document on file. The technical specification was prepared, transmitted to Scott Specialty Gas, and added to the Cycle 17A procurement documentation QA record package prior to the end of the audit (CDA #1). The Work Plan was determined to be adequate, satisfactorily implemented and effective.

5.1.3 Document Control

The audit team reviewed the ANL CMT ACL procedures SOP: ACL-033, *Document Control*, and SOP: ACL-022, *Personnel Signature and Initials Record*. Controlled and current versions of ACL QA documents are accessible via the ACL internet Home Page to ANL-E employees who have been screened to have access to these documents. Superseded hardcopy and electronic versions are archived and made inaccessible for general use. A concern was identified when signed hardcopies of procedures SOP: ACL-158, Revision 04, SOP: ACL-159, Revision 04, SOP: ACL-168, Revisions 0 and 05, and SOP: ACL-169, Revision 03 were not on file. Except for Revision 0 of SOP: ACL-168, the signed copies were found and were placed in the respective binders, and a memo to file was generated to document the reason why Revision 0 of SOP: ACL-168 was not on file (CDA #2). The audit team also verified that the personnel signature and initial list was maintained and revisions to SOPs ACL-169 and -168 were completed using the guidelines of CMT30-0362, *Preparation and Processing of Standard Operating Procedures*. This area was determined to be adequate, satisfactorily implemented and effective.

5.1.4 Quality Assurance Records

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-228, *Disposition of Project Specific Quality Assurance Records*. The audit team verified that the QA records identified in the Attachment 2 of the Work Plan were maintained and controlled. QA records were filed according to the QA Records elements identified in the Work Plan in a two-hour fire rated UL-certified cabinet located in the project record coordinator area. A list of personnel who have access to this file is posted on the cabinet, and the cabinet is locked when not in use. The audit team also verified that corrections to records were made with single line-through, not obliterating the prior entry, and initialed and dated by the originator. This procedure was determined to be adequate, satisfactorily implemented, and effective.

5.1.5 Laboratory Notebooks

The audit team reviewed SOP: ACL-107, *Review of Scientific Notebooks*. The audit team examined the records maintained by the QA Coordinator and verified that all tracking required by the SOP is completed. Objective evidence was obtained showing the effective implementation of the tracking requirements in the procedure. Laboratory Notebooks 1415 and 1787 were reviewed to ensure that the other requirements of the SOP were being implemented. SOP: ACL-107 was determined to be adequate, satisfactorily implemented and effective.

5.1.6 Analytical Sample Preparation

One of the activities conducted by the ANL CMT ACL is verification of HSG PDP samples after they have been prepared. This requires the ANL CMT ACL to prepare one set of PDP canisters and analyze them as though they were samples of unknown concentration. The procedure for preparation of gas sample aliquots for analysis is SOP: ACL-159, *Preparation of Samples for Determination of Headspace - Volatile Organic Compounds (VOCs) in SUMMA® Canisters*. Interviews with the analyst, and review of objective evidence including Laboratory Notebook 1787, demonstrated effective implementation of the procedure. This procedure was determined to be adequate, satisfactorily implemented and effective.

5.1.7 PDP Sample Verification Analyses

As stated above, one of the activities conducted by the ANL CMT ACL is the verification of HSG PDP samples after they have been prepared. This requires the ANL CMT ACL to prepare one set of PDP canisters and analyze them as though they were samples of unknown concentration. The procedure for sample analysis is SOP: ACL-168, *Analytical Method for the Determination of Volatile Organic Compounds in SUMMA® Canisters using Gas Chromatography/Mass Spectrometry*. This procedure was reviewed, prior to the on-site audit, to assess

technical adequacy. Review comments were transmitted to ANL-E, many of which were successfully addressed by the laboratory in a draft revision of the procedure. During the on-site audit, calibration and calibration verification of the GC/MS analytical system was discussed with, and demonstrated by, the responsible analyst. Processing of QC samples and generation of method performance data were also investigated. In general, the analyst was well trained and experienced in his analytical duties, and the equipment and laboratory were adequate for the work performed. Six concerns were identified in this area, two became CDAs and four became an adequacy CAR. Table 1 of SOP: ACL-169 lists the amount of each standard; however, the last calibration standards made (dated 2/18/03) contained an extra compound (DCE). This was not allowed by the procedure and no documentation was generated in association with the procedure deviation. A memo was generated to explain this event and signed by the analyst, group leader, and QA/QC coordinator prior to the end of the audit (CDA#3). Student - t factor used to calculate Method Detection Limits (MDLs) was incorrect for the number of standards analyzed. The Student - t factor in the in-house generated Excel spreadsheet was changed to the correct value and the MDLs re-calculated. A print out of new MDL values was provided during the audit (CDA#4). CAR 03-066 lists adequacy issues in the area of retention time and retention time window determination, generation of precision and accuracy data, canister storage conditions, and generation and disposition of records. Overall, this procedure was determined to be adequate, satisfactorily implemented, and effective.

5.1.8 Gas Standards Preparation

Procedure SOP: ACL-169, *Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA[®] Canisters*, was reviewed, prior to the on-site audit, to assess technical adequacy. Review comments were transmitted to ANL-E, and all of the review comments were successfully addressed by the laboratory in a draft revision of the procedure. Audit of the procedure included discussion with the analyst and demonstration of critical steps in the process. Although the process used to manufacture the calibration standards was acceptable, the use of stock standards beyond their expiration date was a concern. This was identified in CAR 03-065. Records and documentation associated with the stock standards was reviewed, as was the records generated when the calibration standards were made. The laboratory was well organized and the analyst knowledgeable with regard to the processes used. An observation (Observation #2) was identified with regard to documentation of the conductivity of water used to humidify air, although visually checked by the analyst. It was also recommended (Recommendation #1) that the function of the in-line meter, used to verify the conductivity of water, be periodically checked with an independent conductivity meter. Overall, this procedure was determined to be adequate, marginally implemented, and effective.

5.1.9 Preparation and Distribution of PDP Samples

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-211, *"Preparation and Distribution of Simulated Headspace Gas Samples for the WIPP Headspace Gas Performance Demonstration Program."* This procedure was determined to be adequate and effective. However, the procedure was not being implemented entirely as written. Two concerns were identified during the audit. First, the procedure requires that an inventory of all HSG PDP canisters used by the program be maintained in an ACL-approved serialized notebook. In practice, only lists of the canisters distributed in a given cycle are maintained in the notebook. This provides no documentation for canisters owned by the program that are not in use during a cycle. The second concern was that the procedure requires that certificates of analysis received from the gas supplier be signed (or initialed), dated and labeled with the words "Received by ANL." These markings document that a receipt inspection was performed for the procured gasses. None of the certificates of analysis reviewed had the required notations on them. CAR 03- 067 was issued to document these two procedural deviations.

5.1.10 Canister Cleaning

The audit team reviewed the ANL CMT ACL procedure SOP: ACL-158, *"SUMMA® Canister Cleaning."* This procedure was determined to be adequate and effective. However, the procedure was not being implemented entirely as written. Three concerns were identified, two became CDAs and one became a CAR. The Laboratory Notebook #1415 which is maintained to document the canister cleaning activities is also used to document the receipt of returned PDP canisters, but did not indicate where the PDP canisters returned in one of the shipments came from. The shipment records were reviewed to determine where the canisters came from and noted on the laboratory notebook (CDA#5). The same laboratory notebook had entries, documenting cleaning batches, where the canister selected for cleaning certification check analysis was not indicated. The analytical records were reviewed to determine which canisters had been analyzed to certify each cleaning batch and noted on the appropriate notebook pages (CDA#6). The procedure listed the use of a Bourdon type pressure gauge to monitor zero air pressure reading between 0 and 345 kPa (0 and 50 psig) and HPLC grade de-ionized water to humidify the air. In practice, a pressure gauge that read in millimeters of mercury (mm Hg) and water that can not be documented as HPLC grade (rather the water is produced using a NANO pure water system) were used. While use of these items do not impact the effectiveness of the process, they did not revise the procedure to reflect the actual practice. CAR 03- 068 was issued to document this procedural deviation.

6.0 CORRECTIVE ACTIONS REPORTS, OBSERVATIONS, RECOMMENDATIONS, AND CORRECTED DURING THE AUDIT

6.1 Corrective Action Reports

6.1.2 CARs Initiated as a Result of CBFO Audit A-03-18:

The following four CARs, initiated as a result of Audit A-03-18, have been transmitted to the ANL-E under separate cover. A brief description of each CAR is provided below.

6.1.2.1 CBFO CAR 03-065

The standards used for preparation of calibration standards are used beyond their manufacturer's expiration date (for example, ALM030407 and ALM023835 expired in 2001 but were used to make calibration standards on 2/18/03).

6.1.2.2 CBFO CAR 03-066

Adequacy Issues with SOP: ACL-168, Rev. 5:

- A. The initial calibration section (8.4.1) does not describe how retention time and retention time windows are determined.
- B. The procedure does not require generation of method precision and accuracy data.
- C. The procedure does not describe what records are generated by this procedure, nor how records are dispositioned.
- D. The procedure does not address the holding temperature requirement for VOCs SUMMA® canister; hence, there was no documentation for monitoring the temperature in the area for storage of PDP and calibration canisters.

6.1.2.3 CBFO CAR 03-067

SOP: ACL-211, Rev. 4 requires that a log of the location and shipment and content of all canisters used in the HSG PDP be maintained in a serialized notebook; however, only those canisters shipped in a given cycle is recorded in a serialized notebook. The same procedure also requires that receipt inspection be documented by signing (or initialing) and dating the certificate of analysis with a statement "Received by ANL." The certificates of analysis associated with the calibration standards or PDP samples, did not have the statement "Received by ANL."

6.1.2.4 CBFO CAR 03-068

SOP: ACL-158, Rev. 4 requires the use of the Bourdon style pressure gauge to monitor zero air pressure reading between 0 and 345 kPa and HPLC grade water on the

canister cleaning manifold. They used a pressure gauge that measures in mm Hg and water that is purified using the NANO pure system.

6.2 Observations

The audit team identified two conditions that, if left uncorrected, could lead to future conditions adverse to quality. These conditions are reported as Observations, and are being provided to cognizant ANL-E management for consideration.

Observation #1

The Work Plan implies that all personnel involved in the HSG PDP activities read all of the documents that are listed in the Attachment 1 of the Work Plan. Only the Technical Lead for the ANL-E HSG PDP activities has read the QAPD and the HSG PDP Plan. The Work Plan should be revised to be more explicit as to the training requirements for each employee.

Observation #2

The conductivity of water used to humidify air is not documented, although visually checked. This water humidifies air used to clean the standard preparation manifold and must be ASTM Type I and II quality.

6.3 Recommendation

Recommendation #1

It is recommended that the function of the in-line meter, used to verify the conductivity of water, is periodically checked with an independent conductivity meter.

6.4 Deficiencies Corrected During the Audit (CDA)

CDA #1

The Cycle 17A procurement documentation contained no technical specification document as required by the Work Plan for Headspace Gas PDP Sample Preparation, Verification and Distribution, Rev. 7. The technical specification was prepared, transmitted to Scott Specialty Gas, and added to the Cycle 17A procurement documentation QA record package.

CDA #2

Signed hardcopies of procedures SOP: ACL-158, Rev. 04, SOP: ACL-159, Rev. 04, SOP: ACL-168, Revisions 0 and 05, and SOP: ACL-169, Rev. 03 were not on file. The signed copies were found and placed on the respective binders and a memo to file was generated to document the reason why Revision 0 of SOP: ACL-168 was not on file.

CDA #3

Procedure ACL-159, Rev. 03, Table 1 lists the amount of each standard. However, the last calibration standards made (dated 2/18/03) contained an extra compound (DCE). This was not allowed by the procedure and no documentation was generated in association with procedure deviation. A memo was generated to explain this event and signed by the analyst, group leader, and QA/QC coordinator prior to the end of the audit.

CDA #4

Student - t factor used to calculate Method Detection Limits (MDLs) is incorrect for the number of standards analyzed. The Student - t factor in the in-house generated Excel spreadsheet was changed to the correct value and the MDLs re-calculated. Print out of new MDL values was provided during the audit.

CDA #5

One notebook entry documenting the receipt of returned PDP canisters, did not indicate where they came from as required by SOP: ACL-158. The shipment records were reviewed to determine where the canisters came from and noted on the notebook.

CDA #6

Several of the notebook entries, documenting cleaning batches, did not indicate which of the canisters was selected for cleaning certification check analyses as required by SOP: ACL-158. The analytical records were reviewed to determine which canisters had been analyzed to certify each cleaning batch and these notations were applied to the appropriate notebook pages.

7.0 ATTACHMENTS

Attachment 1: Personnel Contacted During the Audit

Attachment 2: Procedures Reviewed During the Audit

PERSONNEL CONTACTED DURING THE AUDIT

PERSONNEL CONTACTED				
NAME	TITLE/ORG	PRE AUDIT MEETING	CONTACTED DURING AUDIT	POST AUDIT MEETING
Boparai, Amrit	Project Manager CMT/ACL	X	X	X
Gaziano, Diane	Deputy Director CMT/ACL	X		X
Kalensky, Mike	Scientific Associate, CMT/ACL	X	X	X
Riel, Roberta	QA Representative, CMT/ACL	X		X
Streets, Elane	QA/QC Coordinator	X	X	X

ANL-E CMT ACL PROCEDURES AUDITED IN A-03-18

NUMBER	PROCEDURE NUMBER/REVISION	TITLE
1.	SOP: ACL-022, R1	Personnel Signature and Initials Record
2.	SOP: ACL-033, R6	Document Control
3.	SOP: ACL-107, R7	Review of Scientific Notebooks
4.	SOP: ACL-158, R4	SUMMA® Canister Cleaning
5.	SOP: ACL-159, R4	Preparation of Samples for Determination of Headspace-Volatile Organic Compounds (VOCs) in SUMMA® Canisters
6.	SOP: ACL-162, R1	Qualification and Training of Analytical Chemistry Laboratory Analysts
7.	SOP: ACL-168, R5	Analytical Method for the Determination of Volatile Organic Compounds in SUMMA® Canisters Sampling Using Gas Chromatographic/Mass Spectrometry
8.	SOP: ACL-169, R3	Preparation of Volatile Organic Compound Calibration Standards for use with SUMMA® Canisters
9.	SOP: ACL-211, R4	Preparation and Distribution of Simulated Headspace Gases Samples for the WIPP Performance Demonstration Program
10.	SOP: ACL-228, R3	Disposition of Project-Specific Quality Assurance Records
11.	No Number/R7	Work Plan for Headspace Gas Performance Demonstration Program Sample Preparation, Verification and Distribution